FORM PTO 1390	ATTORNEY'S DOCKET NUMBER		
(REV. 11-98) U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	OYJALO-008		
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US)	U.S. APPUCATIONNO_(if kgown_sep.37 CER 1.5)		
CONCERNING A FILING UNDER 35 U.S.C. 371	US APPLICATION NO. (If known, see 37 CER 15)		
INTERNATIONAL APPLICATION NO INTERNATIONAL FILING DATES	PRIORITY DATE CLAIMED		
PCT/F199/00696 24 August 1999	24 August 1998		
TITLE OF INVENTION BLEACHING OF MEDIUM CONSISTENCY HIGH SHEAR MIXING	PULP WITH OZONE WITHOUT		
APPLICANT(S) FOR DO/EO/US Rolf DE VOS, et al.			
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/L	S) the following items and other information		
1. X This is a <b>FIRST</b> submission of items concerning a filing under 35 L	ISC 371		
2. This is a SECOND or SUBSEQUENT submission of items concert	ning a filing under 35 U S C 371		
3. X This is an express request to promptly begin national examination	procedures (35 U S C 371 (f))		
4. X The US has been elected by the expiration of 19 months from the p	priority date (PCT Article 31)		
5. X A copy of the International Application as filed (35 U S C 371 (c)(2	?))		
a sattached hereto (required only if not transmitted by the Intern	ational Bureau)		
b X has been communicated by the International Bureau			
c s not required, as the application was filed in the United States	Receiving Office (RO/US)		
6. An English language translation of the International Application as filed (35 U S C 371 (c)(2))			
7. X Amendments to the claims of the International Application under PCT Article 19 (35 U S C 371 (c)(3))			
a are attached hereto (required only if not communicated by the International Bureau)			
b have been communicated by the International Bureau			
c have not been made, however, the time limit for making such amendments has NOT expired			
d X have not been made and will not be made			
8. An English language translation of the amendments to the claims (c)(3))	under PCT Article 19 (35 U S C 371		
9. X An oath or declaration of the inventor(s) (35 U S C 371 (c)(4)) (Unexecuted)			
10. An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U S C 371 (c)(5))			
Items 11. to 16. below concern document(s) or information included:			
11. X An Information Disclosure Statement under 37 CFR 1 97 and 1 98	w/PTO-1449, 5 references		
12. An assignment document for recording A separate cover sheet in compliance with 37 CFR 3 28 & 3 31			
is included  13. A FIRST preliminary amendment			
A SECOND or SUBSEQUENT preliminary amendment			
14. A substitute specification			
15. A change of power of attorney and/or address letter			
16. X Other items or information			
Copy of International Application as published			
Copy of First Written Opinion Copy of Client's Letter of October 25, 2000 in Response to Written	Opinion with Amended Claims		
Copy of International Preliminary Examination Report with Annexe	s		
Copy of International Search Report			

EXPRESS MAIL LABEL NO. <u>EL646757362US</u>
DATE: <u>February 20, 2001</u>

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## BLEACHING OF MEDIUM CONSISTENCY PULP WITH OZONE WITHOUT HIGH SHEAR MIXING

#### Field of the invention

The invention relates to a method for bleaching medium consistency pulp with an ozone-containing gas. In particular, the invention relates to the proper utilization of the very fast reaction of ozone, by providing efficient but pulp-preserving mixing immediately on introducing a substantial amount of ozone into the pulp.

### Background of the invention

A number of methods for the bleaching of pulp with ozone is known in the art. These methods have developed towards carrying out the bleaching stage with medium consistency pulp, i.e. having a consistency of about 7 - 16 per cent.

Generally, ozone bleaching of medium consistency (MC) pulp according to current practice can be described as ozone generation followed by compression before introducing the ozone containing gas into the the MC pulp flow. The gas-liquid-fiber suspension is vigourously treated in one or several high shear mixers before the suspension is lead to a bleach tower. The ozone may be introduced at several points along the pulp stream. Vent gases must be treated because of excess ozone carried over.

The principle described may be a result of the application of oxygen bleaching methods. Oxygen, however, operates at a much slower rate, and the temperatures used are significantly higher than those employed in ozone bleaching.

Typical and frequent problems arise from the difficulty to keep the suspension uniform. Segregation into two-phase flow easily occurs, and the ozonisation rate drops significantly (to 1 or even 0.1 % of its optimum rate) This is a dominant problem, which may be reduced by using a higher quality ozone, resulting in less gas void and consequently less need for vigorous mixing. A typical solution in the present state of the art is the use of more than one mixer. This does not, however, eliminate the problem, and by applying more shear forces to the pulp, the strength properties of the resulting product are severely affected.

A basic problem with such mixers is the short residence time, and if mixing time is increased, undesired backmixing may occur.

After leaving the mixers, the gas-pulp suspension soon segregates into two-phase flow having a relatively small gas-liquid interface per unit volume. The chemical consequences of this are low capacity and a non-uniform bleaching result. Obvious evidence of this phenomenon is the significant ozone surplus often remaining after the bleaching stage, representing both a hazard and an economical loss.

### Description of the prior art

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A pulp bleaching method comprising introduction of high pressure ozone in a carrier gas into a pulp stream with vigorous mixing and subsequent removal of carrier gas is disclosed in, e.g. EP-A 511 433. The major issue of this document is the removal of gas from the pulp after injection into the mixer; the reaction is said to take place essentially within ten seconds in a vertical reaction vessel situated immediately following the fluidizing mixer. Gas at about 10-13 bar containing about 3-10 % ozone by weight (6.8 vol %) is used. Preferably, the gas-pulp mixture is carried in a horizontal path following the vertical reaction step to effect separation of the large amount of carrier gas involved.

Austrian patent application 2203/92 describes a method wherein medium consistency pulp is treated with an ozone-containing gas comprising more than 120 g O<sub>3</sub> / normal m<sup>3</sup> gas (5.6 vol %) whereby the gas is introduced as fine bubbles with a low differential pressure (preferably less than 1 bar). It is considered that using gas with a high ozone content, a sufficient amount of ozone is can be suspended into the gas to achieve the desired bleaching. Further, AT 2203/92 discloses the use of mixers with or without fluidisation effects, and of an ozone reaction stage subsequent to the mixing stage, as well as additional ozone addition stages with degassing stages in between. Characteristically, the highly concentrated ozone is introduced in static mixers at several points, possibly removing the inert carrier gas (normally oxygen) between stages, and the final reaction between ozone and fiber takes place in a bleach reactor, typically of the traditional upflow tower type.

A common feature of several other publications disclosing ozone bleach processes for medium consistency pulp is the use of fluidizing mixers in connection with the injection

of ozone-carrying gas, and the use of subsequent, relatively extended reaction stages and gas separation.

In chemical process terms, MC ozonisation can be described as ozone molecules in a gas phase that must be transported to the vicinity of the fiber and react with the fiber or other substrates. The ozone must diffuse through the gas-liquid interface, through the liquid to the fiber. The applied mixing affects the size and the relative velocity of the gas bubbles, and also the amount of fiber-liquid interface. The rate limiting step completely dominating the interaction of ozone with the fiber material is the transport of ozone through the gas-liquid interface. The gas-liquid transfer rate in a given volume is heavily dependant on the bubble size, i.e. gas-liquid surface area m<sup>2</sup> gas/m<sup>3</sup> suspension, and on the partial pressure of ozone. Other rate limiting steps, like diffusion in the fiber material itself, are determined by the nature and the consistency of the pulp, which is dominantly affected by the temperature.

Due to its dependency on mass transfer, the reaction rate of ozone is, theoretically and empirically, first order.

Consequently, efficient process solutions must be characterized by that

- the residence time distribution (RTD) must follow a plug-flow pattern (in contrast, backmixing commonly occurs in mixers), which requires special reactor geometry to avoid backmixing e.g appropriate turbine and baffles.
- mean residence time in transfer/mixer/reactor must match transport and reaction times for complete conversion of ozone; consequently reactor diameter, shape and rotation rate of a possible turbine must match flow rate.
- all ozone should be introduced in one step.

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The high gas void, i.e. the low concentration of ozone generated by most present ozone generators, limits the possibilities to improve the situation. Reduced gas void in subsequent generations of ozone generators will reduce the need for mixing and reduce energy requirements as well as the size of the equipment. Higher ozone concentrations will also increase the ozonisation rate.

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#### Disclosure of the invention

According to the method of the present invention, high-concentration, high pressure ozone is introduced into the pulp line, whereby conditions approaching plug flow are achieved, a high concentration of ozone is reached with a mass transfer area in the suspension which is sufficient for effective delignification.

According to one aspect of the present invention, the ozone is introduced using effective injection nozzles providing for the efficient dispersion necessary for obtaining a uniform distribution as well as sufficient mass transfer area to overcome the rate-delimiting mass transfer treshold present in methods according to the prior art. Thus, the need for fiber-destroying high shear fluidizing mixers is removed.

According to another aspect of the present invention, a dynamic low to medium intensity mixer is provided in the pulp stream immediately downstream of the ozone injection site. Such a mixer delivers to the pulp stream amounts of energy which are well below fluidization energies, and does not mechanically affect the fiber.

With the aid of recent technology, as disclosed in e.g. Swedish Patent Application 9502339-6, ozone with a concentration of up to 18-20 % by volume may be generated. References to concentrations as high as 300 g O<sub>3</sub>/Nm<sup>3</sup> have been made in prior art publications (e.g. EP-A-426 652, priority 30.10.1989), but such concentrations have not been technically feasible until recently. Using a high ozone concentration (300 g per m<sup>3</sup> and higher) and at high pressure (10 bars and higher) together with proper injection technique, the reaction between ozone and fiber is allowed to take place at such a rate that the subsequent use of an upflow bleach tower is not necessary. The gas pressure is obtained by using precompressed oxygen, optionally mixed with other gases or liquids (e.g. argon) to maintain a suitable conductivity for ozone generation.

Oxygen is the most common carrier gas used for ozone. Highly concentrated ozone is usually considered an explosion hazard. As the ozone generating technology has developed, the accepted limit for stable oxygen-ozone mixtures has been repeatedly pushed upwards, and it appears that no absolute concentration limit for the safe handling of ozone has yet been established. Thus, use of very high ozone concentrations may yet be possible, which further facilitates use of methods according to the present invention. According to the present invention, the concentration of ozone in the gas introduced to

the pulp stream is sufficient for achieving bleaching without any fiber-destroying mechanical impact.

The initial distribution of highly concentrated ozone into the pulp is of importance, for the selectivity, as the carbohydrate component itself may be attacked by ozone if exposed for an extended time. The absence of backmixing, as may occur in high shear mixers, and the presence of plug flow conditions counteract this phenomenon.

### Description of preferred embodiments

Figure 1 shows a comparison between the changes in reaction rates against time in a prior art ozone pulp bleaching process using a medium consistency mixer, and a process according to the present invention.

### Example 1

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Ozone-carrying gas having a pressure of about 15 bar and an ozone concentration 14 % by volume is introduced into a medium consistency pulp line carrying 1000 tons/day via a collar of radially arranged nozzles. Preferably, the nozzles are arranged to direct the gas radially into the pulp flow, essentially in a direction perpendicular to the pulp flow. A number of nozzles sufficient for distributing the gas evenly must be used. On this production scale, 186 nozzles with an inlet diameter of maximum 1 mm may be used. A sufficient mean residence time (10-40 seconds) must be allowed before any other disturbing action to the pulp.

### Example 2

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A medium intensity (low-shear) mixer is adapted into the pulp stream of the previous example, preferably immediately following the gas injection site. The mixer turbine is preferably a double or multiple screw with blade angles and rotation rate balanced to maintain the plug flow residence time distribution (RTD) and giving good radial mixing efficiency. The center blade has a steeper angle than the outer screw blade. Alternatively, porous metal injector devices for introduction of ozone can be arranged peripherically or on the turbine.

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Figure 1 shows a comparison between a system employing a traditional medium consistency mixer with a very high capacity for a short interval dropping rapidly to zero, compared to a system according to the invention with a moderately high capacity kept constant for a long period. The dotted line represents state-of-the-art traditional medium consistency mixer technology. The first, steep section shows the effect of the mixer with high reaction and uniform distribution. The low rate section shows the effect of the corruption of the gas-suspension interface. The reaction takes place with a nonuniform distribution and the pulp is mechanically stressed by high shear mixing.

The solid line represents a system according to the invention. Throughout the process, a moderately fast reaction is taking place in a mildly stressed pulp and with a uniform distribution of ozone.

Table 1 shows a comparison in numbers between a typical conventional MC bleaching system, a state-of-the-art system and a system according to the present invention.

Table 1

		Conventional	Modern	Present invention
Calculus Base	Units			
Pulp production	ton OD/day	1000	1000	1000
Consistency	%	10	10	10
Ozone pressure	bar	9	9	15
Ozone concentration	w%	10	14	20 1 14 = 270
	vol%	7	10	14 = 270
Ozone charge (3-5)	kg/ton OD pulp	5	5	5
Ozone generator	kg/h	208	208	208
Ozone volume flow	m³/s			0,0146
Nozzle diameter	m			0,001
Number of nozzles				186
Process				
Process temperature	°C	40	40	40
Process pressure	bar	7	7	15
Pulp Flow	ton OD pulp /h	42	42	42
Volume Flow	m³/h <b>MC pulp</b>	375	375	375
Ozone gas charge	m <sup>3</sup> /h at actual press.	234	165	53
Gas void *	%	38	31	12
<b>E</b> quipment		Ozone compres-	Ozone com-	No ozone com-
· •		sor	pressor	pressor
		1-3 mixers	1+ mixers	No mixer
		Bleach tower	Bleach tower	Small bleach
				reactor

<sup>\*</sup> Note: Gas void is proportional to process problems

- 1. Method for bleaching of cellulose pulp having medium consistency without using high-shear mixers, comprising the introduction into the pulp stream via radially arranged injection devices of a stream of ozone-containing gas generated from pressurized oxygen or a mixture of pressurized oxygen with at least one gas or liquid and having an ozone concentration of at least about 20 % by weight.
- 2. A method according to claim 1, wherein the introduction of ozone is carried out at a pressure of at least 10 bar.
- 3. A method according to claim 1 or 2, wherein said ozone-containing gas is introduced via at least two nozzles adapted to direct the gas into the pulp stream.
  - 4. A method according to claim 3, wherein said nozzles are adapted to direct the gas in a direction essentially perpendicular to the pulp stream.
  - 5. A method according to any claim 1-4, wherein following gas injection the pulp stream is fed to a dynamic low to medium intensity mixer.
  - 6. A method according to any claim 1-5, wherein ozone-carrying gas is introduced by means of porous metal injector members.

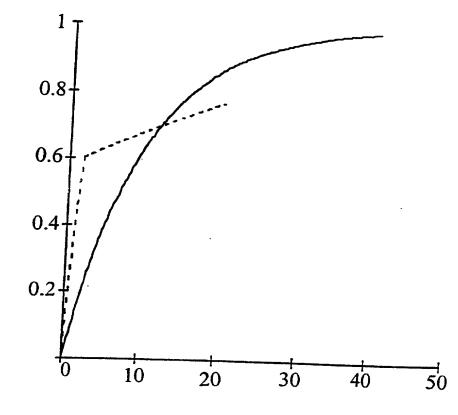
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AMENDED SHEET

### Conversion of ozone



Time, s

FIG. 1

### DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION

ATTORNEY'S DOCKET NO.: OYJALO 3.3-008

As a below-named inventor, I hereby declare that:

My residence, mailing address and citizenship are as stated below next to my name;

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

BLEACHING OF MEDIUM CONSISTENCY PULP WITH OZONE WITHOUT HIGH SHEAR MIXING the specification of which

is attached hereto

☑ was filed on August 24, 1999 as United States Application Number or PCT International Application Number PCT/F199/00696 and was amended on October 25, 2000 (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56

I hereby claim foreign priority benefits under Title 35, United States Code, § 119(a)-(d) of any foreign application(s) for patent or inventor's certificate or § 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor's certificate, or any PCT international application having a filing date before that of the application on which priority is claimed:

filing date before that of the application on which priority is claimed:			
PRIOR FOREIGN APPLICAT	ION(S)		
COUNTRY	APPLICATION NUMBER	DATE OF FILING (month, day, year)	PRIORITY CLAIMED
Finland	981808	August 24, 1998	YES ⊠ NO 🗆
77 E			YES NO NO
2 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1			YES NO
LISTING OF FOREIGN APPLIC	ATIONS CONTINUED ON PAGE 3 HE	REOF YES NO	
hereby claim the benefit under Title 35, United States Code, § 119(e) of any United States provisional application(s) listed below:  Application Number: Filing Date:			
Application Number: Filing Date:			
hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s), or § 365(c) of any PCT international application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is get disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.			
ES. Parent Application Serial Number: Parent Filing Date: Parent Patent No.:			
U.S. Parent Application Scrial Num	ber Parent Filin	g Date:	arent Patent No.:
PCT Parent Number,	Parent Piling	g Date:	
LISTING OF US APPLICATIONS CONTINUED ON PAGE 3 HEREOF 🔲 YES 🔯 NO			

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following registered practitioner(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith: Customer Number 000530

DIRECT ALL CORRESPONDENCE TO: Customer No. 000530

### DECLARATION - Page 2

### ATTORNEY DOCKET NO. OYJALO 3.3-008

I hereby declare that all attatements made herein of my own knowledge are true and that all statements made on information and belief are balieved to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereos.

Full name of sole or first inventor (even name, family name): ROLF DE VOS

Full dame of sole of first inventor (given name, family name); ROII DE VO	3
Inventor's signature Aut (C)	Date 6/04/7
Residence: Landvetter, Sweden Citizenship: Sweden Mailing Address: Backa Gård 54, S-438 36 Landvetter, Sweden	
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Third Inventor's signature	Date
Residence: Citizonship: Mailing Address:	
Full name of fourth joint inventor, if any (given name, family name):	
Fourth Inventor's signature	Date
Residence: Citizenship: Mailing Address:	
Full name of fifth joint inventor (given name, family name):	
Fifth Inventor's signature	Date
Residence: Citizenship: Mailing Address:	
rull game of sixth joint inventor, it any (given name, family name):	
Sixth Inventor's signature	Date
Residence: Citizenship: Mailing Address:	
Full name of seventh joint inventor, if any (given name, family name):	
Seventh Inventor's signature	Date
Residence: Citizenship: Mulling Address:	
Full name of eighth joint inventor, if any (given name, family name):	
Eighth Inventor's signature	Date
Residence: Citizenship: Mailing Address:	
Additional inventors are being named on separately numbered sheats attach	od bereto.

### DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION

ATTORNEY'S DOCKET NO.: OYJALO 3.3-008

fis a below-named inventor, I hereby declare that:

My residence, mailing address and citizenship are as stated below next to my name;

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

BLEACHING OF MEDIUM CONSISTENCY PULP WITH OZONE WITHOUT HIGH SHEAR MIXING the specification of which

is attached hereto

was filed on <u>August 24, 1999</u> as United States Application Number or PCT International Application Number PCT/F199/00696 and was a needed on <u>October 25, 2000</u> (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment specifically referred to above.

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PRIOR FOREIGN APPLICATION COUNTRY	APPLICATION NUMBER	DATE OF FILING (month, day, year)	PRIORITY CLAIMED
Finland	981808	August 24, 1998	YES 🔯 NO 🗌
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LISTING OF FOREIGN APPL	CATIONS CONTINUED ON PAGE 3 H	EREOF YES NO	
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application designating the United S not disclosed in the prior United S Code 5 112 Legipopulates the	Fitle 35, United States Code, \$120 of any States of America, listed below and, inset tates or FCT international application in a duty to disclose information which is available between the filing date of the particular contents.	ofar as the subject matter of each he manner provided by the first p material to patentability as defi	of the claims of this application is paragraph of Title 35, United State ned in Title 37, Code of Federa
U.S. Parent Application Serial Nu	mber: Parent Fili	ig Date:	Parent No.:
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PCT Parent Number:	Parent Pilin	g Date	
LISTING OF US APPLICATION	S CONTINUED ON PAGE 3 HERBOF:	🗆 yes 🖾 no	
POWER OF ATTORNEY: As a transact all business in the Patent	named inventor, I hereby appoint the follow and Trademark Office connected therewith	wing registered practitioner(s) to	prosecute this application and to

DIRECT ALL CORRESPONDENCE TO: Customer No. 000530

Full name of sole or first inventor (given name, family name): Rolf DE VOS

# DECLARATION - Page 2

#### ATTORNEY DOCKET NO. OYJALO 3.3-008

It hereby declars that all statements made herein of my own knowledge are true and that all statements made on information and belief are the believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful fulse statements may jeopardize the validity of the application or any patent issued thereon.

Inventor's signatu	ire	Date
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Full name of secon	d joint inventor, if any (given name, family name) Panu TIKKA	1 1 1 1 1 7
Second Inventor's	s signature	Dute
Residence. Raun Mailing Address:	na, Finland Chizmship: Finland  (sokatu 38, FIN-26100 Rauma, Finland  /AULAAJA-BULKEY  joint inventor, if any (given name, family name):	
Third Inventor's	zignature	Date
Residence: Mailing Address:	Citizenship:	
Full name of fourt	h joint inventor, if any (given pame, family name).	
Fourth Inventor	signature	Bate
Residence: Mailing Address:	Citizenship:	
Full name of fifth	joint inventor (given name, family namo):	
Fifth Investor's	signature	Date
Residence: Mailing Address.	Citizenship:	
Full name of sixth	o joint inventor, if any (given name, family name):	
Sixth Inventor's	signature	Date
Residence: Mailing Address:	Citizership:	
Full name of seve	enth joint inventor, if any (given name, family name)	
Seventh Invento	r's signature	Date
Residence: Mailing Address:	Çitizenship:	
Full name of eigh	th joint inventor, if any (given name, family name):	
Eighth Inventor	's signature	Date
Residence: Mailing Address:	Citizenship:	
Additional in	ventors are being named on separately numbered sheats attached hareto.	